

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 1 of 14

PURPOSE

To record the receipt of samples and the analyses requested, to assign sample identification numbers, and to store samples under appropriate conditions.

SCOPE

All samples sent to AXYS for analysis must be correctly logged-in prior to proceeding with analytical testing. Log-in procedures include the following steps:

- receipt and inspection of samples,
- verification that sample acceptance criteria are satisfied,
- assignment of a contract number and AXYS ID numbers,
- generation of a LIMS Login Chain-of-Custody Report,
- labelling of sample containers,
- storage of samples,
- documentation of above procedures.

Samples are received by the Sample Receiving Chemist or Purchasing and Receiving personnel. Sample log-in is done by the Sample Receiving Chemist only. In the case of absence or after-hours delivery, a Project Chemist or a Laboratory Services Chemist, trained in sample receiving procedures, may perform the sample receiving and log in tasks.

Project Chemist or Sample Receiving Chemist reviews and approves all sample login information and then releases the samples for analysis.

PROCEDURES

Often, AXYS has advance notice of the shipment of samples. A Project Chemist or Client Services Manager notifies the Sample Receiving Chemists and Purchasing and Receiving personnel of an expected delivery. They provide the following information as available:

- Client name and contact number
- Name of the courier and tracking (waybill) number OR the name of the individual making the delivery and a contact number
- Approximate date of delivery

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 2 of 14

- Number and type of sample
- Any special instructions

Monday – Friday Deliveries

The Sample Receiving Chemist contacts the couriers in the morning to track any samples due to arrive that day, or if the samples have not arrived by their expected delivery time. If the delivery is to be later than the close of business they contact the Project Chemist or the Client Services Manager. They will contact the client regarding the delay and make arrangements for after hours delivery (if necessary).

Saturday Deliveries

A list of contact numbers is posted in the Sample Receiving Area and in the Purchasing and Receiving Office.

1. For Saturday deliveries, the Sample Receiving Chemist tracks any expected samples with the courier company on the previous Friday, and ensures that the samples are tagged by the courier for Saturday delivery. If the delivery is delayed until Monday, they contact the Project Chemist or Client Services Manager by phone at their home and/or by pager. They will attempt to contact the client regarding the delay and they send an e-mail message to these individuals about the Monday delivery.
2. The Sample Receiving Chemist tracks the samples on Saturday morning to ensure there are no delays due to customs or paperwork. If the expected shipment does not arrive by 3:00 pm, the Sample Receiving Chemist contacts the courier service to track the shipment. They contact the Project Chemist or Client Services Manager by phone at their home and/or pager. If the sample is still expected that day, they will make arrangements for after hours delivery. If the shipment is lost or delayed until Monday, they will contact the client and attempt to locate the sample with the courier service.
3. If the Sample Receiving Chemist is unable to contact the Project Chemist or Client Services Manager, and they are unable to stay to receive the after hours samples, they contact one of the Laboratory Services Supervisors or designee.

It is essential someone be at AXYS to receive the samples.

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 3 of 14

Receipt of After Hours Deliveries

When the shipment arrives:

1. The Sample Receiving Chemist completes the LIMS Sample Receiving Log and records the receiving information.
2. The Sample Receiving Chemist stores the samples under the conditions specified by the analytical method, in the appropriate storage location as specified in Table 1 "Summary of Storage Facilities" of SLA-004, or in the contract.
3. The Sample Receiving Chemist sends an e-mail message to the Project Chemist and Client Services Manager noting the delivery of samples and the date and time the samples were received.

Samples submitted to the laboratory must be properly processed before the samples can be handled in the laboratory. The first page of an "AXYS Sample Receiving Record" (FSA-015) must be completed for every shipment of samples received by AXYS. This form records the condition of samples upon receipt and indicates whether or not the samples meet AXYS' Sample Acceptance Criteria. Pages 1 and 2 of the AXYS Sample Receiving Record satisfy the U.S. Environmental Protection Agency (USEPA) requirements for the use of their Form DC-1, and are used to receive and log in samples from the USEPA. Contract requirements may specify the use of specific sample log-in forms. In these cases, the specific form is also to be completed when logging in samples.

I. SAMPLE RECEIPT INSTRUCTIONS

Refer to the appropriate Sample Acceptance Criteria and Sample Storage Requirements section of the relevant method document when carrying out the following procedures. Use the project notes to determine the correct method document(s). Record the information described below on the appropriate section on page 1 of the AXYS Sample Receiving Record and, if necessary, on a client-specified form. If a client-prepared Chain of Custody form accompanies the samples, record relevant information (e.g., received time and temperature) on the Chain of Custody form as well. For samples received from the USEPA, also complete page 2 of the AXYS Sample Receiving Record. Samples may have specific handling and storage instructions specified by the client. In these cases, client or contract specifications supersede those listed below, and are to be adhered

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 4 of 14

to. The person logging in the samples is to include their name on the AXYS Sample Receiving Record.

1. When a shipment of samples arrives at the laboratory, record the courier waybill number (if present), the client name, and the date and time received on the Sample Receiving Record (FSA-015) in LIMS. The LIMS generates and records a unique Receipt Number for the shipment and records the initials of the person receiving the shipment. Record the condition of the shipping container, noting any damage.
2. Measure the temperature of the samples upon receipt by either (1) inserting a thermometer into the liquid of a cooler temperature indicator bottle, if present, or (2) placing a thermometer deep inside the container, next to a sample container. Use a calibrated thermometer ($\pm 1^{\circ}\text{C}$) with a measurable range of at least -10 - 50°C . Allow the thermometer to equilibrate for 3-5 minutes before recording the temperature. Record the temperature.

Compare the temperature reading with the required sample receipt temperatures as specified in the method Sample Storage Requirements or in the Project Notes. If samples do not meet the temperature requirements, make a note of this on the receiving record. Notify the Client Services Manager or Project Chemist who will contact the client about sample deficiencies.

3. Review the Chain-of-Custody or other documents which accompany the samples and record the information included with the documents. Record whether preservative has been added or the addition of preservative has been requested. Log-in any samples requiring preservation as "PRETREAT" and provide details in the "Log-in Info" section in the LIMS. Release the samples in the LIMS to the Lab Services group for pre-treatment. If preservation has been requested, notify the Sample Preparation Lab Manager. Attach these documents to the AXYS Sample Receiving Record (or client specified log-in form). If there is no sample log or documentation with the samples, notify the Client Services Manager or Project Chemist. They will contact the client for instructions. Place the samples in temporary storage until the proper documentation is available.
4. Record the presence or absence of custody seals. If the seals are present, record the location of the seals, the condition (intact or broken) of the seals, and the custody seal numbers, if present. If the seals have been broken, notify the Client Services Manager or Project Chemist immediately. They will contact the client for instructions.

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 5 of 14

5. Visually inspect the contents of the client's cooler(s) or box(es). Record the condition of sample containers, and verify that the samples meet the sample requirements for the requested method(s) (refer to method-specific Sample Storage Requirements). Record any problems or discrepancies. For additional procedures regarding the preservation of aqueous samples, refer to Steps 5 and 6 in the Sample Handling Section of this SOP. If any sample jars are broken, or samples are damaged, or any sample acceptance criterion is not met, notify the Client Services Manager or Project Chemist immediately. They will contact the client for instructions. If a damaged or broken sample can be salvaged, contact Laboratory Services Group personnel who are responsible for transfer of the sample to another container. Note the damage to the sample and the transfer operation on the Receiving Record. In the LIMS "Comments" section record any sample deficiencies, damage or transfers to another container.
6. Cross-reference the sample label information with the client sample log. If a Chain-of-Custody form is shipped with the samples, use this to cross-reference the samples. If there are discrepancies, notify the Client Services Manager or Project Chemist who will contact the client for instructions. Record deficiencies on the Receiving Record.
7. If sample tags are present, cross-reference the sample tags against the sample container labels and against the Chain-of-Custody or client sample log. If there are discrepancies, notify the Client Services Manager or Project Chemist who will contact the client for instructions. Record deficiencies on the Receiving Record.
8. Sign the Sample Receiving Record when complete.
9. If samples are from a new client or new contract, assign a new contract number from the LIMS Account List. Inform the Client Services Manager of the new contract number. If a contract number already exists for the client's current work, then use the existing contract number. Write the contract number on the client correspondence, Chain-of-Custody, and receiving records. The following system is in place for assigning contract numbers:

Commercial Clients and Government Contracts:

Contract numbers between 2000 and 2999 and between 9600 and 9999 were assigned to commercial clients and these are still used. As of 01 April 2000, sequential contract numbers starting at 4000 are assigned to new commercial clients and government contracts.

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 6 of 14

QA/QC Analyses:

Contract numbers are assigned to interlaboratory studies and performance evaluation samples. These studies are assigned sequential contract numbers starting at 3000.

- 10 Carry out the Sample Log-in procedures on the day the samples are received. If this is not possible, place the samples in appropriate temporary storage as follows:
 - a) Put a label on the shipping container. The label should read "Not Logged-in" and have client's contract number on it.
 - b) Put the container in the appropriate storage area.
 - c) Record the and the storage location of the samples on the appropriate documentation accompanying the samples. File in the "Work-in-Progress" slot in Sample Receiving area and note the date received and location of samples on the white board in the Sample Receiving area.

II SAMPLE LOG-IN

A Sample Receiving Chemist carries out login procedures. A Project Chemist or a Sample Receiving Chemist must approve all sample logins. Approval occurs by signing the LIMS Login Chain of Custody Report. The Chemist is to ensure that all client-specific storage and handling procedures are met as required.

1. Access the LIMS, as described below, to obtain the log-in ID for a client's shipment of samples.
 - Log on to Seedpak,
 - Under "Sample Management", open "Log-in",
 - Select "New" for the LIMS assigned sample number.
2. The LIMS automatically assigns a unique AXYS Sample ID number to each sample. The AXYS Sample ID is of the form LNNNN-N and is composed of the Login ID (the letter "L" followed by four digits) followed by sequential numbers, one for each sample, starting at 1. The "Login ID" (e.g. L1234) is a unique identifier for each set of sample(s) submitted by an individual client. Write the AXYS Sample ID number beside the corresponding client sample number on the appropriate client correspondence, including a client-supplied Chain-of-Custody, any client specific form, and on page 2 of the Receiving Record, if necessary.

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 7 of 14

3. Using the LIMS, log-in each sample under the client's contract number and, if required, under the client's "Project" or "Catalogue" number. As appropriate, enter the following information into the LIMS:
 1. the AXYS Sample ID number (generated automatically by the LIMS),
 2. the matrix type (pulp, effluent, tissue, sediment, etc.),
 3. client sample identification (obtain from the client supplied documentation and/or the container label),
 4. the date received
 5. the Receipt Number
 6. the container type and number of containers,
 7. the collection date and time,
 8. the identity of the sampler
 9. the storage location,
 10. the assigned Project Chemist
 11. the due date, which defaults to 30 days, or as specified by contract requirements,
 12. the analysis requested (e.g., dioxins, PAH, moisture, lipids),
 13. the Media Return LIMS identification number,
 14. Comments - any other relevant information or instructions, including sample deficiencies, or if any samples need to be returned,
4. Label each sample using self-stick labels and affix labels to samples. Labels may be handwritten or generated by the LIMS once the log-in process has been completed. Handwritten labels must be prepared using a waterproof felt-tip marker. LIMS-generated labels include the following information:
 - AXYS Sample ID number,
 - the client sample identification (obtained from client documents and/or the sample label),
 - the date the samples were received,
 - the Permit Number if applicable (tissue or imported sediments
 - the client Account Number

As a minimum, handwritten labels must include the AXYS Sample ID number.

In addition, when an individual sample is submitted in several sample containers, the containers are to be labelled "1 of 2", "2 of 2", etc. Currently this is done by hand.

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 8 of 14

5. A Project Chemist or Sample Receiving Chemist, a person who did not log in the samples, reviews the LIMS Login Chain of Custody Report, and makes any necessary corrections. In the case of routine analyses such as "Precursors", the Sample Receiving Chemist who logs-in the samples may approve LIMS Login Chain of Custody Report by signature, and "release" the project in the LIMS to make the samples available (in the LIMS) for laboratory analysis.
6. For most projects, a Project Chemist generates Project Notes (specific Work Instructions for the laboratory), and then uses the LIMS to release the samples for analysis.
7. When samples have been released by the Sample Receiving Chemist or by a Project Chemist it is the responsibility of the Sample Receiving Chemist to photocopy the approved Login Chain of Custody Report and distribute copies to the appropriate Lab Manager(s) responsible for the requested analyses.
8. Give the original approved LIMS Login Chain of Custody Report, the documentation accompanying the samples, and any other client correspondence and instructions to the Client Services area for filing in the appropriate client file.
9. If it is necessary to make changes to a LIMS Login Chain of Custody Report after distribution, print the revised LIMS Login Chain of Custody Report and re-distribute. If possible, identify the changes made.

III SPECIAL INSTRUCTIONS

Sample Log-in

1. If a sample has been sent as a replacement or back-up sample, assign the sample a new number. Note in the LIMS the ID of the previous sample (e.g., Replacement for sample "...").

Injection Ready Sample Extracts for Instrumental Analysis

1. Receive injection ready sample extracts in autosampler vials, sent to AXYS for GC/MS analysis, according to procedures in Section I. Inspect the vials for damage, including verifying the seal around cap. Report deficiencies to the Client Services Manager to discuss with the client.

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 9 of 14

2. If a damaged sample requires further action, e.g., transfer to another autosampler vial, contact an Instrumental Analytical Chemist, who carries out the required procedure.
3. Log the samples into the LIMS according to the procedures in Section II. Place a label with the AXYS ID number and client sample number on each vial. It is the responsibility of the Sample Receiving Chemist to place these samples in a Workgroup in the LIMS in order to initiate the analysis process.
4. Approve the LIMS Login Chain of Custody Report. Give the copies of the documentation to the Instrumental Analysis Manager and the original documents to the Client Services area. Also give the extracts to the Instrumental Analysis Manager. Store the extracts in the Laboratory 1 refrigerator freezer until just prior to analysis.

Sample Handling/Preservation

Operations involving Handling or Preservation of water samples must be performed as soon as possible. It is essential that personnel in the Laboratory Services are immediately notified of the arrival of these samples by telephone, E Mail, and/or personal contact.

1. Wet Pulps/Sludges

Some wet pulps and sludges must be dried prior to analysis. Consult with a Project Chemist regarding drying requirements, as this is a parameter- and project-specific operation. If required, drying of these samples is the responsibility of the Laboratory Services Group.

If drying is required, log-in a LIMS, "PRETREAT" product and notify the Laboratory Services Group of the receipt of wet pulps or sludges. If they cannot be dried immediately, place the pulps in appropriate storage. When the drying procedure is complete, store the dry sample and excess wet sample and update the LIMS with the storage locations.

2. Effluents for Regular Chlorophenol Analysis

Effluent samples must be preserved with ascorbic acid as soon as possible if chlorophenolic analysis is required. This is the responsibility of the Sample Preparation Laboratory.

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 10 of 14

Notify a Project Chemist of the receipt of effluent samples for chlorophenol analysis. The Project Chemist will instruct the Laboratory Services Group of the details of preservation (preservative and amount). A Laboratory Services Chemist adds the preservative and notes the preservation on the sample label and records the preservation and storage of these samples in the LIMS.

3. *Sediments for Chlorophenolic Analysis*

Sediment samples that have standing water must have the pH of the water tested as soon as possible if chlorophenol analysis is requested. This is the responsibility of a Chemist in the Sample Preparation Area. Notify a Project Chemist of the receipt of sediment samples for chlorophenol analysis that have standing water.

Notify the Laboratory and Sample Preparation Area of the receipt of sediment samples for chlorophenol analysis. A Chemist in the Laboratory and Sample Preparation Area tests the pH and makes any necessary pH adjustments according to SLA-013. The adjustments and decantation is recorded on the sample label. Filtration or centrifugation procedures may need to be performed to prevent loss of fine particulate.

4. *Sediments with Standing Water*

It may be necessary to decant standing water from sediment samples prior to storage at -20°C. Filtration or centrifugation procedures may need to be performed to prevent loss of fine particulate. Inform a Project Chemist and the Lab Services group by e-mail if sediment samples have standing water. The Project Chemist will contact the client for instructions.

5. *pH Adjustment of Aqueous Samples*

If the analytical method calls for a pH adjustment, immediately refer to the documentation that accompanies the samples. Determine whether the pH check and adjustment were carried out in the field. If so, indicate that a pH adjustment is not required. If the pH check and adjustment were not carried out or if there is some doubt, indicate that a pH adjustment is required and notify the Laboratory Services Group. A Chemist from the Laboratory Services performs the pH adjustment.

Notify a Project Chemist of the receipt of aqueous samples that require pH adjustment.

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 11 of 14

6. *Residual Chlorine in Aqueous Samples*

If the analytical method requires a check for residual chlorine, immediately refer to the documentation that accompanies the samples. Determine whether a residual chlorine check and adjustment were carried out in the field. If so, indicate that a residual chlorine adjustment is not required. If the residual chlorine check and adjustment were not carried out or if there is some doubt, indicate that a residual chlorine adjustment is required and notify the Laboratory Services Group. A Laboratory Services Chemist performs the check and preservation following SLA-073 or SLA-075 (if required).

Notify a Project Chemist of the receipt of aqueous samples that require residual chlorine check and adjustment.

7. *Composite Samples*

AXYS is often requested to prepare a composite sample from several individual samples. Upon receipt, perform the usual pre-logging inspection as per steps 1 to 4. Unless otherwise instructed by the Project Chemist, log in the samples according to the following procedures:

Each individual sample used in preparation of a composite sample (including samples which may be totally consumed in the preparation of a composite sample) is logged into the LIMS as individual samples and are assigned individual AXYS Sample ID numbers. These samples are to be given a LIMS "COMPOSITE" Analysis Product. Assign the composite sample a unique AXYS ID number (the "client identification" of the composite sample may be specified by the client). The composite sample is to be given a LIMS "HOMOGENIZATION" Analysis Product. Complete all log-in information for the individual and the composite samples as described under "Sample Log-In" above. The label for the Composite sample will later be prepared by the Laboratory Services Chemist and placed on the composite sample container by them when the composite is prepared.

Ensure the LIMS login entry for the Composite sample clearly lists the individual samples making up the Composite. In addition, ensure that each individual sample also lists the identity of the Composite sample that the individual sample will be placed into.

Note that in some cases a client will submit several discrete samples in a single container (e.g. several fish) but will provide instructions to treat this as one sample. In this case, the

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 12 of 14

sample is to be treated as a single sample.

IV. SAMPLE STORAGE

1. Store samples in the appropriate location for the particular matrix type. Refer to the Sample Storage Requirements Table in the specific analytical method for proper storage requirements.

In situations where immediate log-in and method specific sample storage are not possible (i.e. analysis has not been specified, unusual matrix, complex analysis requests and heavy workload and/or time constraints) the following temporary storage conditions may be used.

Matrix	Storage Condition
XAD column (filter + resin) Aqueous samples Stack gas sample trains Wet pulp, sludge Solvent extracts Air (PUF + Filter)	4°C
Solids (e.g. Sed/soil/ash/part.filter) Tissues (including blood, milk) Extracts in autosampler vials	-20°C
Dry pulp Oil Defoamer	Ambient
Wet solid requiring sample pre-treatment or preservation	4°C

V QC CHECKS AND CORRECTIVE ACTIONS

1. Accuracy of login information is independently verified as described in section II-5. This review must be completed within 3 working days of sample receipt. Where errors are discovered the corrections must be made within 1 working day of discovery.

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 13 of 14

2. Follow up verification of any corrections requested to log-in information, including sample label modification, LIMS entry, or supporting documentation changes must be completed by the Project Chemist within 2 days of requesting changes.

AXYS Analytical Services Ltd Standard Operating Procedure

Title: Sample Receipt and Login
Area: Laboratory Procedures

SOP #: SLA-003
Rev. No.: 12
Date: 09-May-2003
Page: 14 of 14

References

QDO-003 Sample Acceptance Criteria
FSA-005 AXYS Chain-of-Custody Form
FSA-015 AXYS Sample Receiving Record
SLA-004 Sample Control Procedure
SLA-013 Homogenization Procedures
SLA-073 Starch/KI Determination of Free Chlorine in Aqueous Samples
SLA-075 Colorimetric Determination of Free Chlorine in Aqueous Samples

Approval:

Dale Hoover, Quality Manager

Date

Dr. Coreen Hamilton, Technical Director

Date